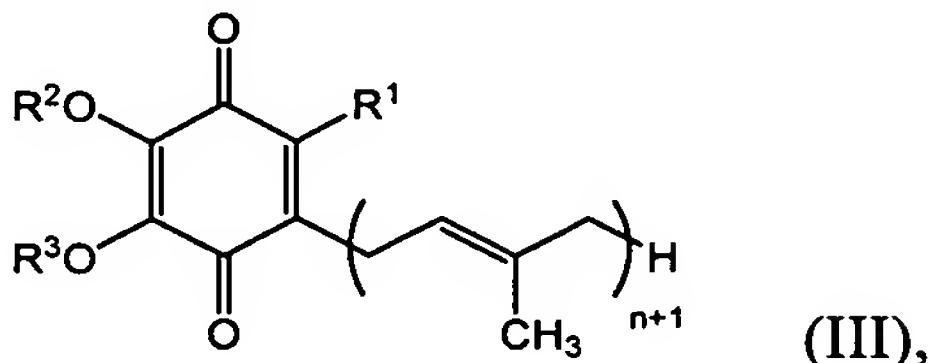


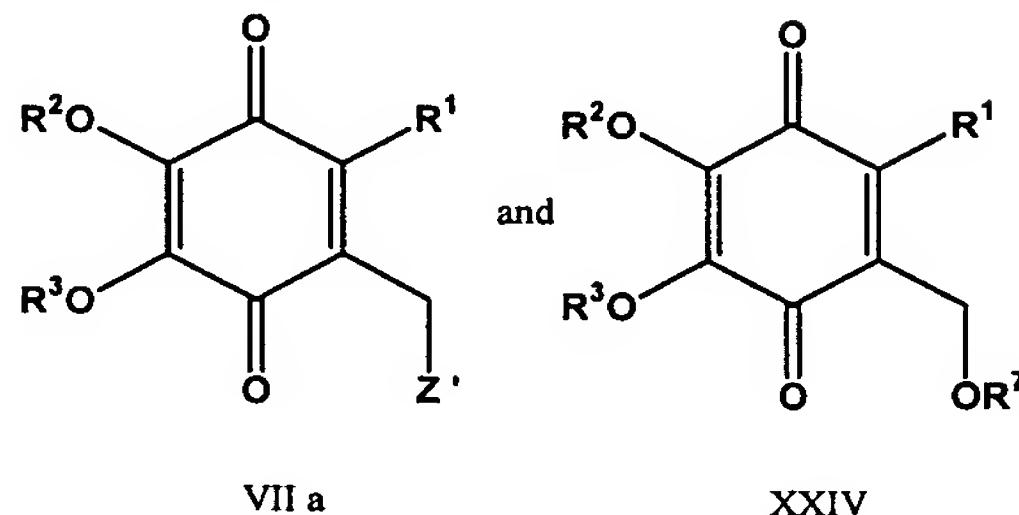
## **WHAT IS CLAIMED IS:**

## 1. A method of preparing a compound of Formula



said method comprising:

4 contacting a compound that is a member selected from:



## 6 in which

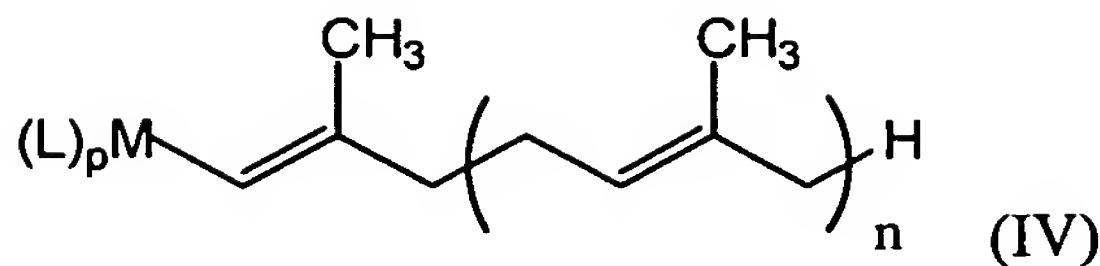
$R^1$ ,  $R^2$  and  $R^3$  are independently selected from substituted or unsubstituted  $C_1$ - $C_6$  alkyl groups;

wherein

each R<sup>9</sup> and R<sup>10</sup> is a member independently selected from substituted or unsubstituted alkyl, substituted or unsubstituted aryl, substituted or unsubstituted heteroaryl and substituted or unsubstituted heterocycloalkyl; and

Z' is a leaving group other than halogen,

20 with a compound having the structure



wherein

each L is independently selected from substituted or unsubstituted alkyl, alkoxy, aryl or aryloxy with 1 to 10 carbon atoms;

M is aluminum;

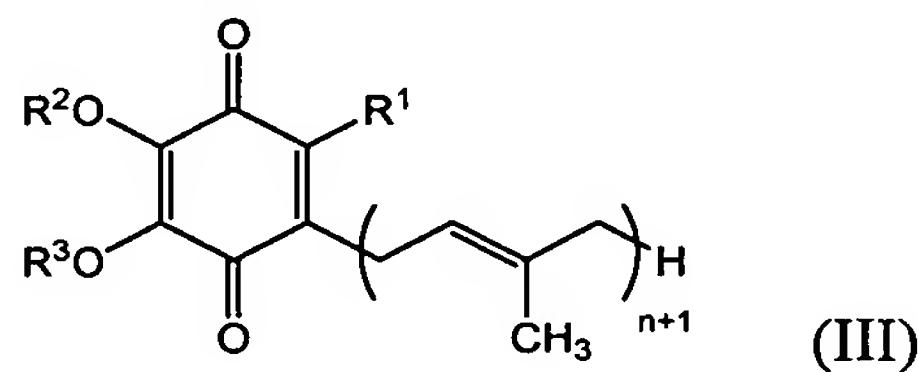
p is 1 or 2

n is an integer from 0 to 19,

in the presence of a coupling catalyst effective at catalyzing coupling between the methylene carbon of the quinone of Formula (VII) a or (XXIV) and the vinylic carbon attached to M,

thus preparing said compound of Formula (III).

2. A method of preparing a compound having the formula:

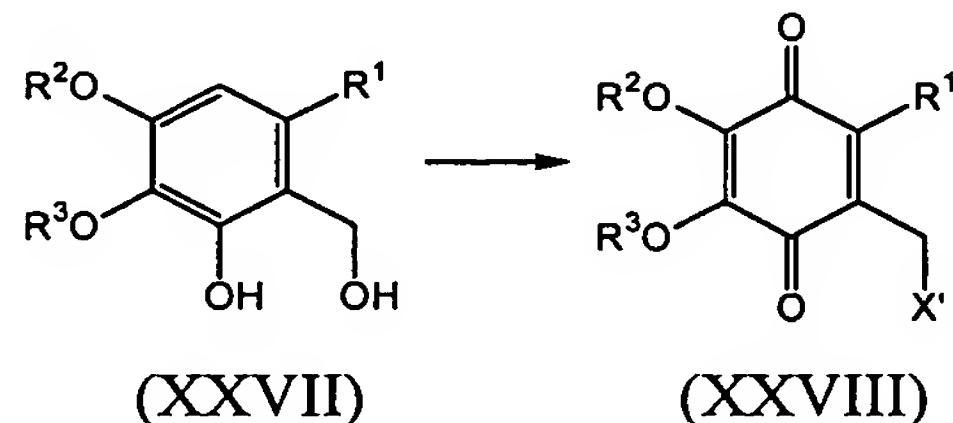


wherein

R<sup>1</sup>, R<sup>2</sup> and R<sup>3</sup> are members independently selected from substituted or unsubstituted C<sub>1</sub>-C<sub>6</sub> alkyl groups; and

n is an integer from 0 to 19, said method comprising:

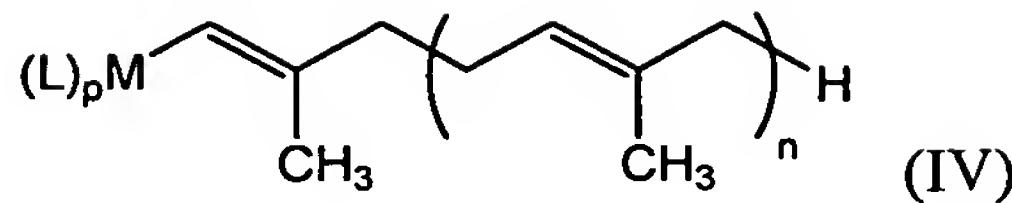
(a) performing the transformation:



wherein

X' is OH or a leaving group; and

(b) contacting the product of (a) with:



14 wherein

15 each L is independently selected from substituted or unsubstituted alkyl,  
16 alkoxy, aryl or aryloxy with 1 to 10 carbon atoms;

17 M is aluminum;

18 n is an integer from 0 to 19;

19 p is 1 or 2;

20 in the presence of a coupling catalyst effective at catalyzing coupling between the  
21 methylene carbon of the quinone of Formula XXVIII and the vinylic carbon  
22 attached to M in Formula (IV)

23 thus preparing said compound of Formula (III).

1           3.     The method according to claim 1 or 2, wherein R<sup>1</sup>, R<sup>2</sup> and R<sup>3</sup> are  
2 methyl.

1           4.     The method according to claim 1 or 2, wherein L is methyl.

1           5.     The method according to claim 2, further comprising, prior to step (a):

2           (c) formylating the compound:



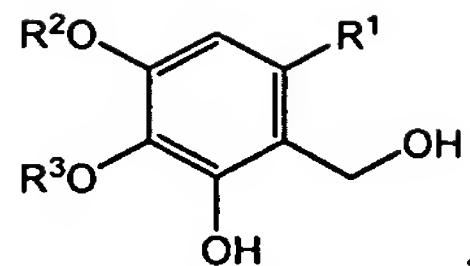
4 forming:



6           (d) demethylating the product of (c), forming:



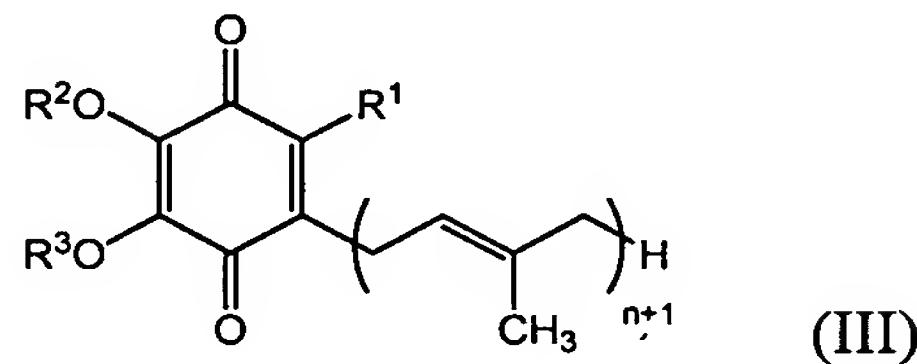
8           (e) reducing the product of (d), forming:



9

(XXXIV)

1           6.     A method of preparing a compound having the formula:



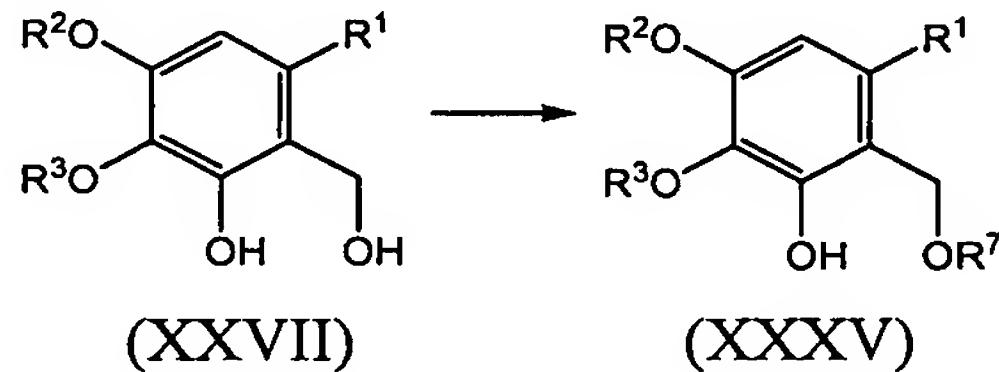
2

3     wherein

4     R<sup>1</sup>, R<sup>2</sup> and R<sup>3</sup> are members independently selected from substituted or unsubstituted  
5     C<sub>1</sub>-C<sub>6</sub> alkyl groups; and

6     n is an integer from 0 to 19, said method comprising:

7           (a) performing the transformation:



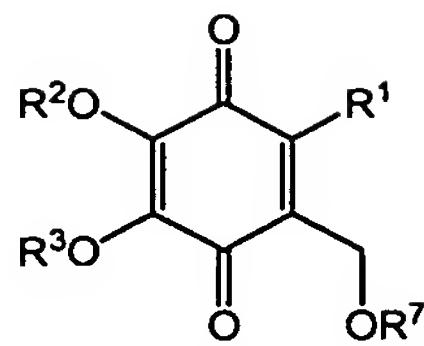
8     wherein

9     R<sup>1</sup>, R<sup>2</sup> and R<sup>3</sup> are members independently selected from substituted or unsubstituted  
10    C<sub>1</sub>-C<sub>6</sub> alkyl groups; and

11    R<sup>7</sup> is selected from H, substituted or unsubstituted alkyl, substituted or  
12    unsubstituted heteroalkyl, substituted or unsubstituted aryl, substituted or  
13    unsubstituted heteroaryl, substituted or unsubstituted  
14    heterocycloalkyl, SOR<sup>9</sup>, SO<sub>2</sub>R<sup>9</sup>, C(O)R<sup>9</sup>, C(O)OR<sup>9</sup>, P(O)OR<sup>9</sup>OR<sup>10</sup>,  
15    P(O)N(R<sup>9</sup>)<sub>2</sub>(R<sup>10</sup>)<sub>2</sub>, and P(O)R<sup>9</sup>R<sup>10</sup>

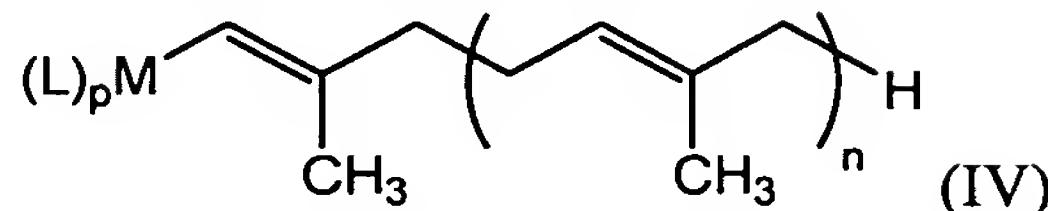
16    wherein each R<sup>9</sup> and R<sup>10</sup> is a member independently selected from substituted  
17    or unsubstituted alkyl, substituted or unsubstituted aryl, substituted or  
18    unsubstituted heteroaryl and substituted or unsubstituted  
19    heterocycloalkyl; and

20    (b) oxidizing the product of (a) to a compound having the formula:



and

(c) contacting the product of (b) with:



wherein

each L is independently selected from substituted or unsubstituted alkyl, alkoxy, aryl or aryloxy with 1 to 10 carbon atoms;

M is aluminum;

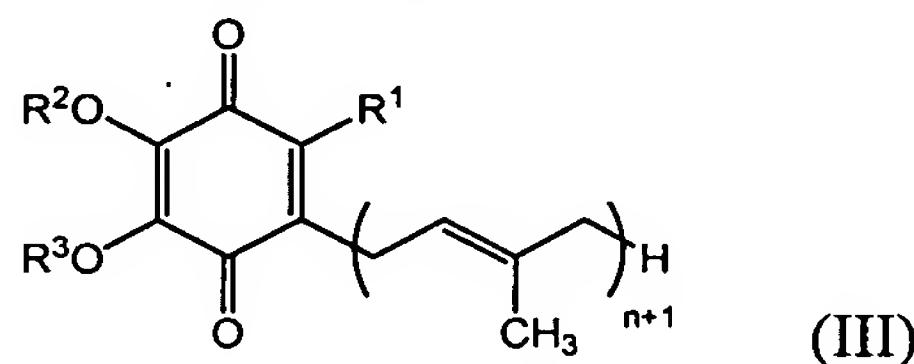
p is 1 or 2;

n is an integer from 0 to 19

in the presence of a coupling catalyst effective at catalyzing coupling between the quinone methylene carbon of the compound of Formula (XXIV) and the vinylic carbon attached to M,

thus preparing said compound of Formula (III).

1           7.       A method of preparing a compound having the formula:

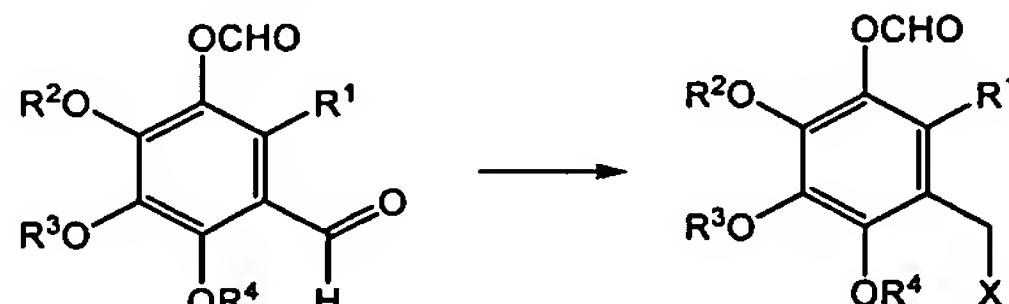


wherein

R<sup>1</sup>, R<sup>2</sup> and R<sup>3</sup> are members independently selected from substituted or unsubstituted C<sub>1</sub>-C<sub>6</sub> alkyl groups; and

n is an integer from 0 to 19, said method comprising:

(a) performing the transformation:



9 (XXIII)

10 (XXXVI) ;

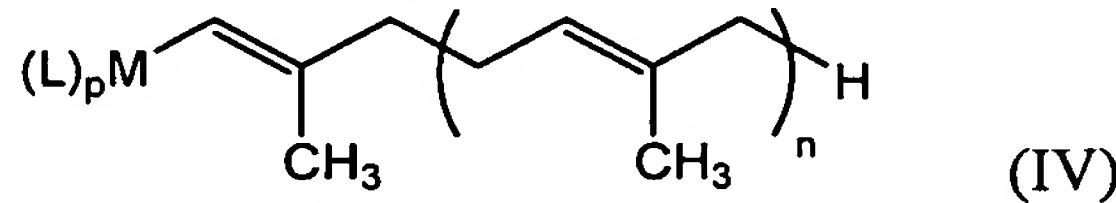
11 wherein

12  $R^1$ ,  $R^2$  and  $R^3$  are independently selected from substituted or unsubstituted  
13  $C_1$ - $C_6$  alkyl groups;

14  $R^4$  is a member selected from hydrogen, substituted or unsubstituted alkyl,  
15 and protecting groups; and

16  $X$  is a leaving group;

17 (b) contacting the product of (a) with:



(IV)

19 wherein

20 each  $L$  is independently selected from substituted or unsubstituted alkyl,  
21 alkoxy, aryl or aryloxy with 1 to 10 carbon atoms;

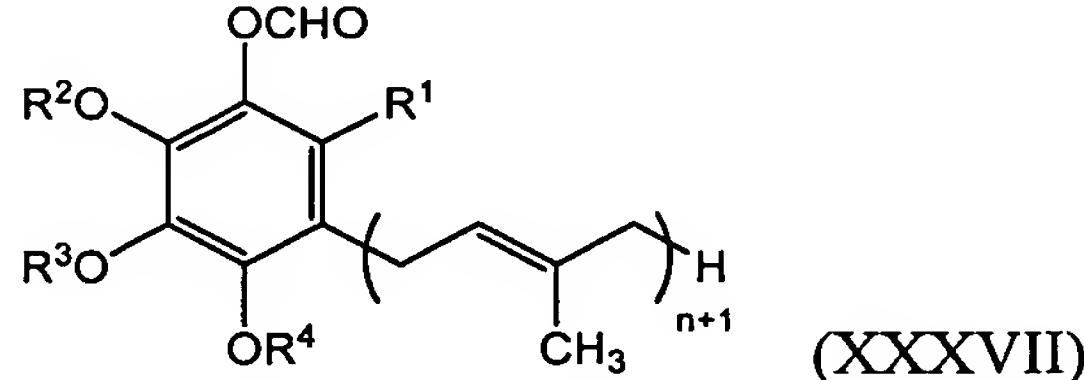
22  $M$  is aluminum;

23  $p$  is 1 or 2;

24  $n$  is an integer from 0 to 19

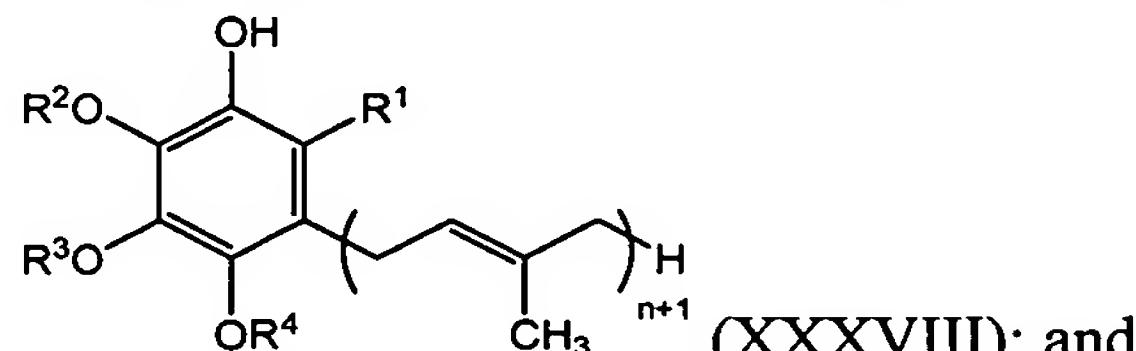
25 in the presence of a coupling catalyst effective at catalyzing coupling between the

26 substituted methylene carbon atom of the compound of Formula (XXXVI) and the  
vinylic carbon attached to  $M$ , forming:



(XXXVII)

28 (c) deprotecting the product of (b), forming:



(XXXVIII); and

30 (d) oxidizing the product of (c),

31 thus forming said compound of Formula (III).

1 8. The method according to claim 1, 2, 6 or 7, wherein said coupling  
2 catalyst comprises a transition metal.

1                   **9.**       The method according to claim **8**, wherein said transition metal is  
2       Ni(0).

1                   **10.**      A method of carboaluminating an alkyne substrate, forming a species  
2       with an alkyl moiety bound to aluminium, said method comprising:

3                   (a) contacting said alkyne substrate with  $(L)_{p+1}M$  and  $x$  molar equivalents of  
4       water or  $R^{20}OH$ , or, when each L is methyl, with  $x$  molar equivalents  
5       of water,  $R^{20}OH$  or methylaluminoxane relative to said alkyne  
6       substrate

7                   wherein

8                    $0 < x < 1$ ;

9                   each L is independently selected from substituted or unsubstituted alkyl,  
10               alkoxy, aryl or aryloxy with 1 to 10 carbon atoms;

11               M is aluminium;

12               p is 1 or 2 and,

13                $R^{20}$  is branched or unbranched alkyl with 1 to 15 carbon atoms, optionally  
14               substituted with 1 to 5 hydroxy substituents,

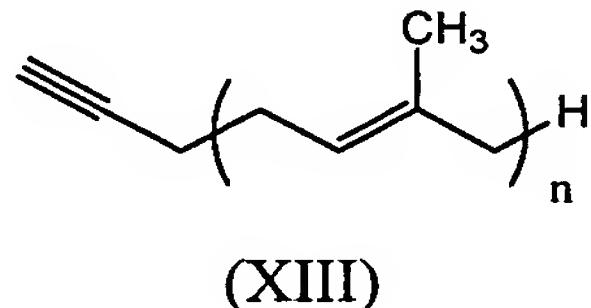
15       thus carboaluminating said alkyne substrate .

16

17

1                   **11.**      The method according to claim **10**, wherein said alkyne substrate is a  
2       terminal alkyne.

1                   **12.**      The method according to claim **11**, wherein said alkyne substrate has  
2       the formula:



1                   **14.**    The method according to claim **10**, said method further comprising  
2 contacting said alkyne substrate with a carboalumination catalyst, in an amount less than one  
3 equivalent relative to said alkyne substrate.

1                   **15.**    The method according to claim **14**, wherein said carboalumination  
2 catalyst is used in an amount of less than 0.2 molar equivalents relative to said alkyne  
3 substrate.

1                   **16.**    The method according to claim **14**, wherein said carboalumination  
2 catalyst is a member selected from zirconium- and titanium-containing species.

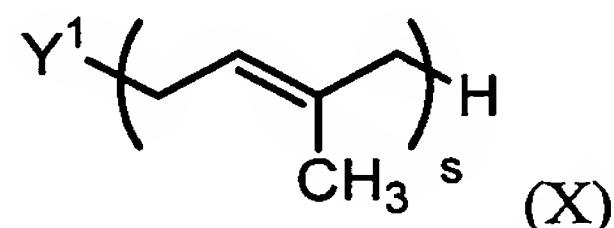
1                   **17.**    The method according to claim **10**, wherein said carboalumination is  
2 conducted in a solvent or solvent mixture comprising at least one non-chlorinated solvent.

1                   **18.**    The method according to claim **17**, wherein said non-chlorinated  
2 solvent is a member selected from trifluoromethylbenzene and toluene.

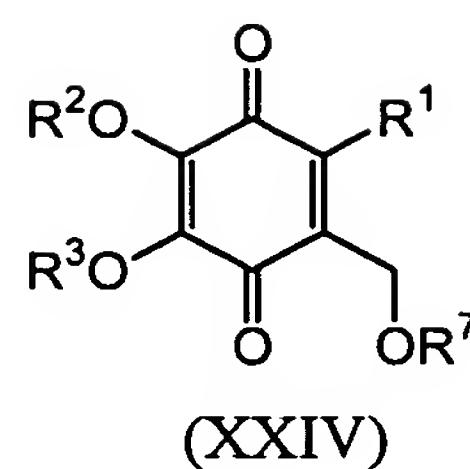
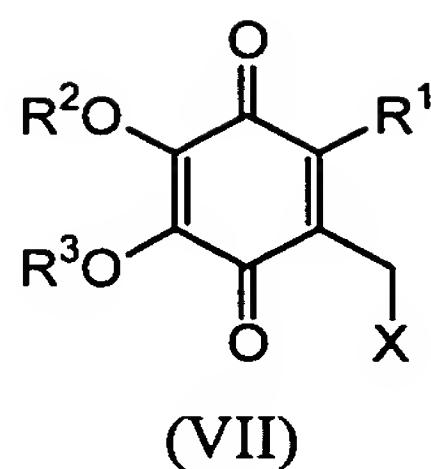
1                   **19.**    The method according to claim **17**, wherein said carboalumination is  
2 conducted in trifluoromethylbenzene or toluene or mixtures thereof.

1                   **20.**    The method according to claim **12**, wherein said alkyne substrate is  
2 produced by:

3                   (a) forming propyne dianion by contacting propyne with a base; and  
4                   (b) combining said propyne dianion with a compound having the formula:



3      Formula (VII) or (XXIV),



in which

$R^1$ ,  $R^2$  and  $R^3$  are independently selected from substituted or unsubstituted  $C_1$ - $C_6$  alkyl groups;

$R^7$  is selected from H, substituted or unsubstituted alkyl, substituted or unsubstituted heteroalkyl, substituted or unsubstituted aryl, substituted or unsubstituted heteroaryl, substituted or unsubstituted heterocycloalkyl,  $SOR^9$ ,  $SO_2R^9$ ,  $C(O)R^9$ ,  $C(O)OR^9$ ,  $P(O)OR^9OR^{10}$ ,  $P(O)N(R^9)_2(R^{10})_2$ , and  $P(O)R^9R^{10}$

wherein

each  $R^9$  and  $R^{10}$  is a member independently selected from substituted or unsubstituted alkyl, substituted or unsubstituted aryl, substituted or unsubstituted heteroaryl and substituted or unsubstituted heterocycloalkyl; and

$X$  is a leaving group,

under conditions appropriate to couple the carboaluminated product of step

(a) in claim 10 with the methylene carbon atom of the compound of Formula (VII) or (XXIV).

1      23.     The method according to claim 22, wherein step (b) is conducted

2     essentially without prior purification of the product of step (a) of claim 10.

1      24.     The method according to claim 22, wherein in step (b) a compound of

2     Formula 13 is contacted with a product of step (a) in claim 10.

1      25.     The method according to claim 24, wherein a compound 13 is used in

2     form of a mixture further comprising a compound of formula 14.

1      26.     The method according to claim 25, wherein the mixture comprising

2 compounds **13** and **14** is used after filtration through an adsorbent medium.

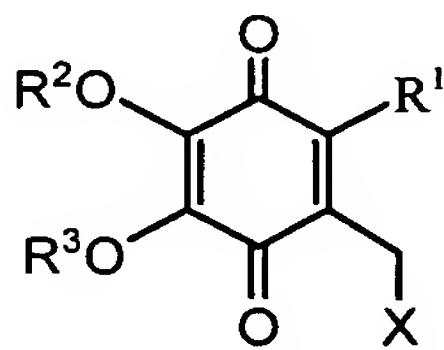
1                   **27.** The method according to claim **26**, wherein said adsorbent medium is  
2                   alumina.

2 said method comprising:

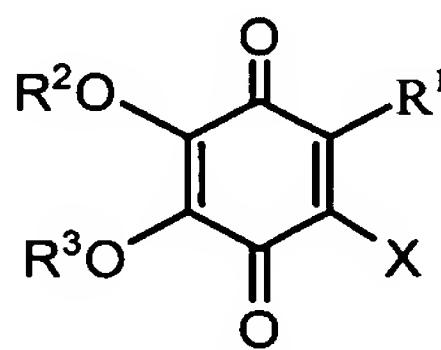
- (a) contacting a reaction mixture comprising said alkyne substrate of Formula (XIII) with an adsorbent medium; and
- (b) eluting said alkyne substrate from said adsorbent medium and collecting said alkyne substrate as a single fraction; and
- (c) submitting the product from step (b) to a carboalumination reaction essentially without further purification,

9 thus carboaluminating said alkyne substrate.

1                   **29.**     A method of separating components of a mixture, said components  
2 comprising a substituted-methylene quinone and a quinone having the formulae:



; and



(VII)

(XXXIX),

5 respectively

6 in which

$R^1$ ,  $R^2$  and  $R^3$  are independently selected from substituted or unsubstituted  $C_1$ - $C_6$  alkyl groups;

X is a leaving group;

10 said method comprising:

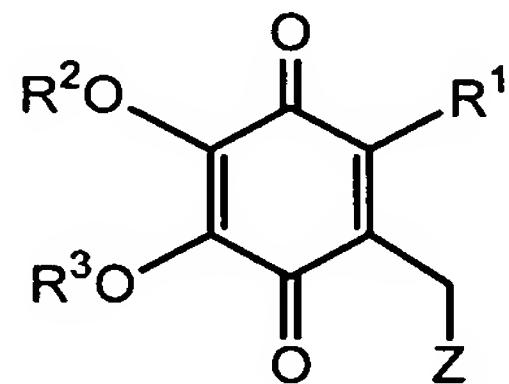
(a) contacting the mixture with a reactive species that selectively binds through a heteroatom to the methylene carbon of said substituted-methylene quinone, displacing said leaving group, producing a charged substituted-methylene quinone; and

15 (b) separating said charged substituted-methylene quinone from said quinone,

thereby separating said mixture.

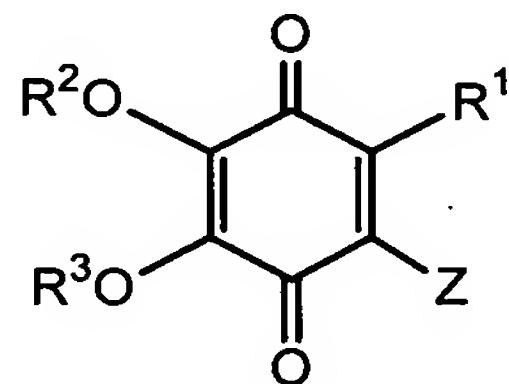
1                   **30.**    The method according to claim **29**, further comprising, contacting the  
2    substituted-methylene quinone with a vinylalane, under conditions appropriate to form a  
3    ubiquinone.

1                   **31.**    A method of separating a substituted methylene quinone and a halo-  
2    quinone having the formulae:



13

; and



14

3                   4    respectively

5                   5    in which

6                   6    R<sup>1</sup>, R<sup>2</sup> and R<sup>3</sup> are independently selected from substituted or unsubstituted  
7    C<sub>1</sub>-C<sub>6</sub> alkyl groups;

8                   8    Z is a halogen;

9                   9    said method comprising:

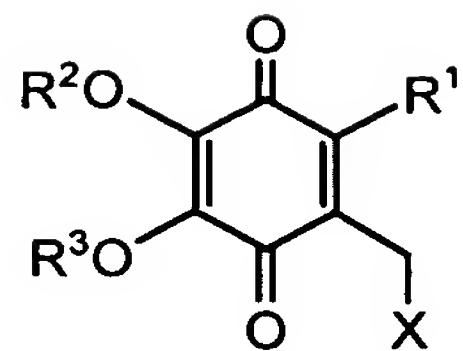
10                  10    (a) contacting said mixture with a reducing agent that selectively reduces the  
11                   11    halo-quinone to a halo-hydroquinone;

12                  12    (b) contacting the product of step (a) with a base, forming an anion of said  
13                   13    halo-hydroquinone; and

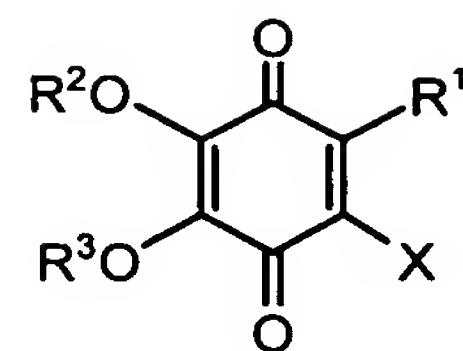
14                  14    (c) separating said anion from said substituted methylene quinone, thereby  
15                   15    separating said mixture.

1                   **32.**    The method according to claim **31**, further comprising, contacting the  
2    said substituted methylene quinone with a vinylalane, under conditions appropriate to form a  
3    ubiquinone.

1                   **33.**    A method of separating a mixture of a substituted-methylene quinone  
2    and a quinone having the formulae:



; and



4 (VII),

(XXXIX)

5 respectively

6 in which

7  $R^1$ ,  $R^2$  and  $R^3$  are independently selected from substituted or unsubstituted

8  $C_1$ - $C_6$  alkyl groups;

9  $X$  is a leaving group;

10 said method comprising:

11 (a) contacting the mixture with a reactive species that selectively binds  
12 through a heteroatom to the methylene carbon of said substituted-  
13 methylene quinone, displacing said leaving group;

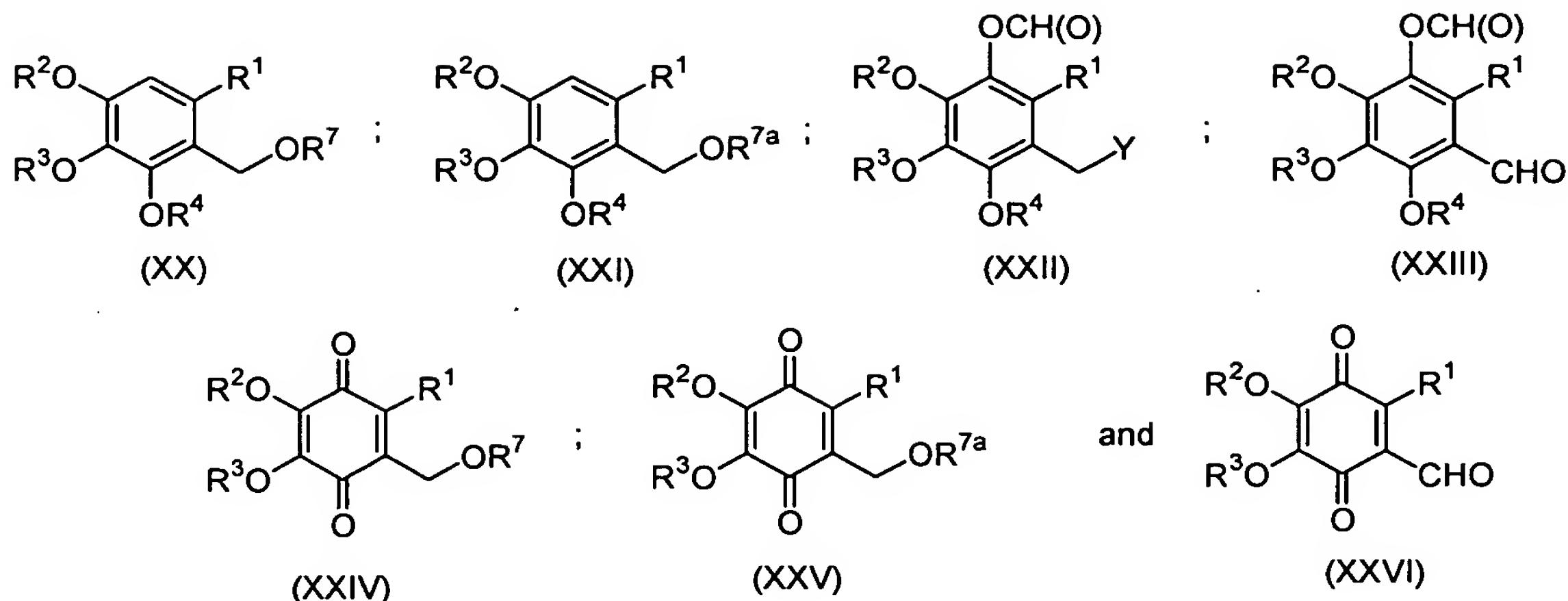
14 (b) separating the product of (a) from said quinone, thereby separating said  
15 mixture.

1 34. The method according to claim 33, wherein said reactive species is a  
2 substituted or unsubstituted  $C_1$ - $C_{20}$  carboxylate.

3 35. The method according to claim 33, wherein said separating is by  
4 chromatography.

1 36. The method according to claim 33, further comprising, contacting the  
2 substituted-methylene quinone with a vinylalane, under conditions appropriate to form a  
3 ubiquinone.

1 37. A compound having a structure that is a member selected from:



2

3 in which

4         $R^1$ ,  $R^2$  and  $R^3$  are independently selected from substituted or unsubstituted  $C_1$ - $C_6$   
5        alkyl groups;

6         $R^4$  is a member selected from H, substituted or unsubstituted alkyl, a metal ion and a  
7        protecting group;

8         $R^7$  is selected from H, substituted or unsubstituted alkyl, substituted or unsubstituted  
9        heteroalkyl, substituted or unsubstituted aryl, substituted or unsubstituted  
10        heteroaryl, substituted or unsubstituted heterocycloalkyl,  $SOR^9$ ,  $SO_2R^9$ ,  
11         $C(O)R^9$ ,  $C(O)OR^9$ ,  $P(O)OR^9OR^{10}$ ,  $P(O)N(R^9)_2(R^{10})_2$ , and  $P(O)R^9R^{10}$   
12        wherein

13        each  $R^9$  and  $R^{10}$  is a member independently selected from substituted  
14        or unsubstituted alkyl, substituted or unsubstituted aryl,  
15        substituted or unsubstituted heteroaryl and substituted or  
16        unsubstituted heterocycloalkyl; and

17         $Y$  is  $OR^{11}$ ,  $SR^{11}$ ,  $NR^{11}R^{12}$ , or a leaving group;

18         $R^{11}$  and  $R^{12}$  are independently selected from H, substituted or unsubstituted  
19        alkyl, substituted or unsubstituted heteroalkyl, substituted or  
20        unsubstituted aryl, substituted or unsubstituted heteroaryl and  
21        substituted or unsubstituted heterocycloalkyl; and

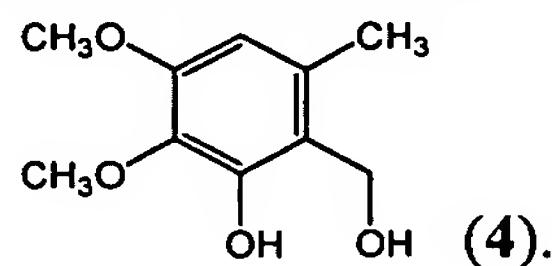
22         $R^{7a}$ , together with the oxygen to which it attached, is a leaving group.

1        38. The compound according to claim 37, wherein  $R^{7a}$  is a member  
2        selected from  $SOR^9$ ,  $SO_2R^9$ ,  $C(O)R^9$ ,  $C(O)OR^9$ ,  $P(O)OR^9OR^{10}$ ,  $P(O)N(R^9)_2(R^{10})_2$ , and  
3         $P(O)R^9R^{10}$

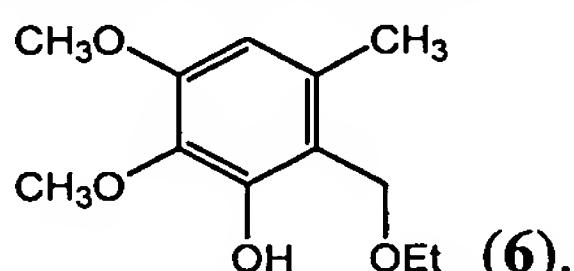
4        wherein

5 each R<sup>9</sup> and R<sup>10</sup> is a member independently selected from substituted or  
6 unsubstituted alkyl, substituted or unsubstituted aryl, substituted or  
7 unsubstituted heteroaryl and substituted or unsubstituted heterocycloalkyl.

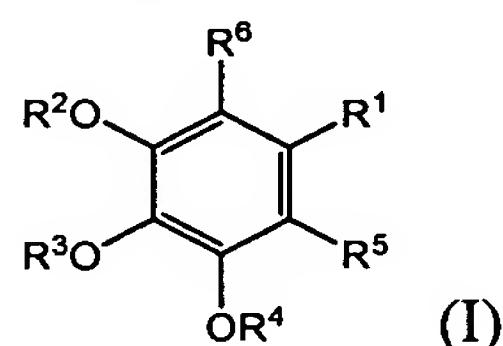
1 39. The compound according to claim 37, having the formula:



1 40. The compound according to claim 37, having the formula:



1 41. A compound having the formula:



3 wherein

4 R<sup>1</sup>, R<sup>2</sup> and R<sup>3</sup> are members independently selected from substituted or unsubstituted  
5 C<sub>1</sub>-C<sub>6</sub> alkyl groups;

6 R<sup>4</sup> is a member selected from hydrogen, substituted or unsubstituted alkyl, and  
7 protecting groups;

8 R<sup>5</sup> is a member selected from branched, unsaturated alkyl, CH(O), CH<sub>2</sub>Y  
9 wherein

10 Y is OR<sup>7</sup>, SR<sup>7</sup>, NR<sup>7</sup>R<sup>8</sup> or a leaving group

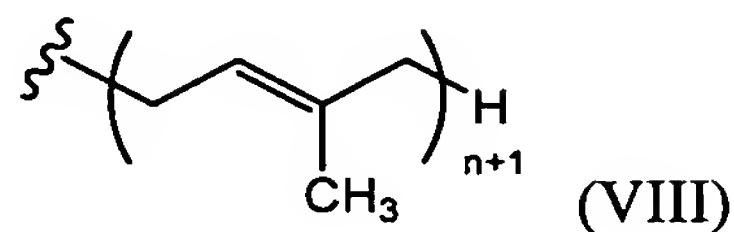
11 wherein

12 R<sup>7</sup> and R<sup>8</sup> are members independently selected from H, substituted or  
13 unsubstituted alkyl, substituted or unsubstituted heteroalkyl,  
14 substituted or unsubstituted aryl, substituted or unsubstituted  
15 heteroaryl and substituted or unsubstituted heterocycloalkyl;  
16 and

17 R<sup>6</sup> is a member selected from OH and OCH(O).

1                   **42.**    The compound according to claim 41, wherein  $R^5$  is a moiety having

2   the formula:

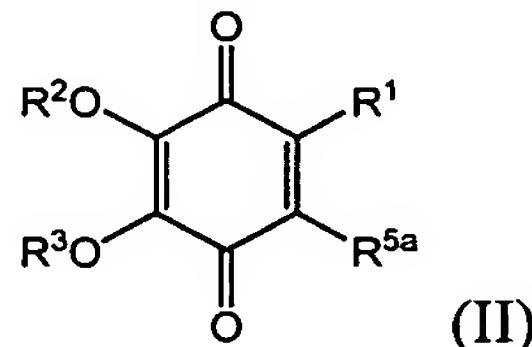


(VIII)

wherein

$n$  is an integer from 0 to 19.

1                   **43.**    The compound according to claim 37, having the formula:



(II)

wherein

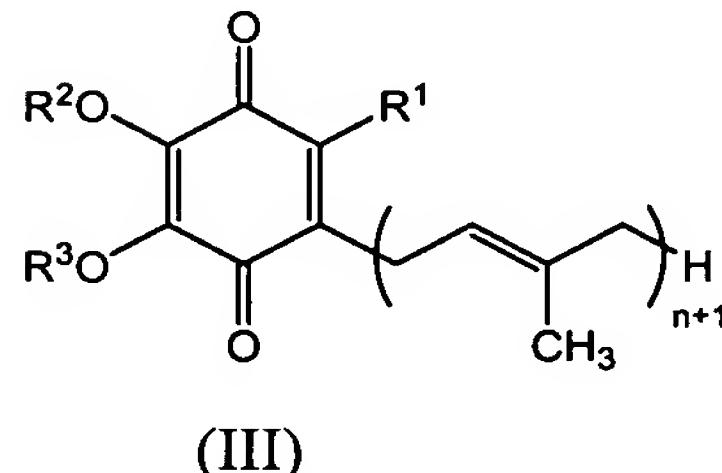
$R^1$ ,  $R^2$  and  $R^3$  are members independently selected from substituted or unsubstituted  $C_1$ - $C_6$  alkyl groups; and

$R^{5a}$  is a member selected from  $CH(O)$  and  $CH_2OR^{7a}$

wherein

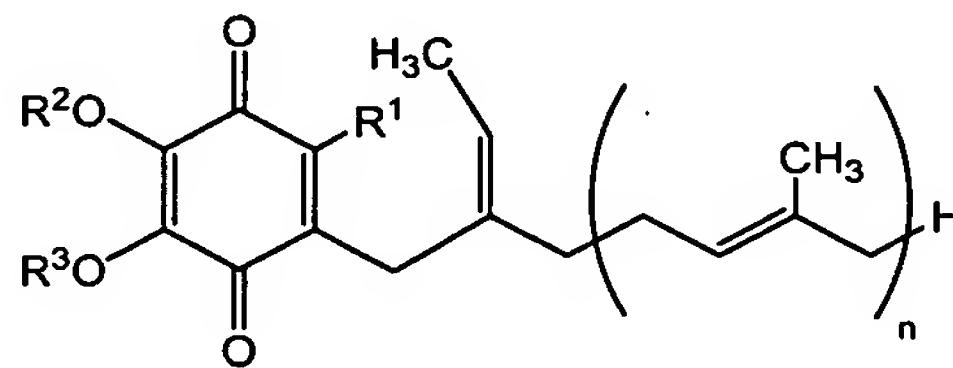
$R^{7a}$  is selected from H and substituted or unsubstituted alkyl.

1                   **44.**    A mixture comprising:



(III)

and



(IX)

wherein

$R^1$ ,  $R^2$  and  $R^3$  are members independently selected from substituted or unsubstituted  $C_1$ - $C_6$  alkyl groups; and

$n$  is an integer from 0 to 19.

1                   **45.**    A mixture according to claim 44, wherein  $n$  is 9.

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1                   **46.**    A mixture according to claim 44, wherein  $R^1$ ,  $R^2$  and  $R^3$  are methyl.

47. A mixture according to claim 44, in which the molar ratio of the compound of Formula (III) to the compound of Formula (IX) is at least 8 to 1.